THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

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NICKEL PLATING TROUBLES AND CURES

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(*An abstract of this paper appeared in Nickel Bulletin, July-August, 1954.*)

In applying the information given to British practice, it should be noted that all the units quoted are U.S. standards. One British gallon equals approximately 1·2 American gallons.

THE troubles that may beset a plating foreman are many and varied, and the possible causes and probable cures now fill many a page in reference books.

It is far better to avoid the troubles, by close control, than to have to cure them. The Chinese thought it expedient to pay a doctor to keep them well, which pay was withheld when illness occurred.

The analogy with plating may not be too obvious, but, logically, control of plating operations by a trained foreman or a laboratory staff affords insurance against troubles that may cause costly rejects or shutdowns.

The following chart, based on the authors' experiences with several hundred installations, covers only nickel plating trouble shooting in a general sense. For more complete and explicit information, the literature already published should be consulted.

NICKEL PLATING TROUBLE SHOOTING CHART

Possible Source of Trouble	Suggested Cure	Possible Source of Trouble	Suggested Cure
1. LACK OF ADHESION (PEELING)			
(*a*) Cleaning Cycle	Cleaners should be tested for concentration, temperature, current density, chromic acid or other contamination. When hexavalent chromium is found, it may be overcome by treatment with sodium hyposulfite ($Na_2S_2O_4$), as outlined by M. M. BECKWITH§. Water rinses should be checked for cleanliness.	(*b*) Lack of acid dip	A proper concentration acid dip and water rinse should be used as final steps before the work is plated.
		(*c*) Current interrupted	Breaking of current should be avoided, as the laminated deposit may not be adherent, especially when the plated ware is bent.

* United Chromium, Inc., Detroit, Mich.
† Editor, *Plating*, Newark, N.J.
‡ Department of Chemistry, Boston College, Boston, Mass.
§ M. M. BECKWITH, *Proc. Amer. Electroplaters' Soc.*, 1941, vol. 29, pp. 80–6.

Possible Source of Trouble	Suggested Cure
(*d*) Repair work	Repair work should be stripped clean before it is replated, or the old nickel surface must be activated. Activation of the work is accomplished in either of two ways. In a cyanide activator, the work is made cathodic for 30 to 60 seconds at 70°–80°F. in a 12-ounce-per-gallon solution of sodium cyanide. At least 6 volts are needed to produce vigorous gassing on the work being activated. Some workers prefer to precede the cathodic treatment with an anodic shot of a few seconds. Activation is also carried out in the Wood chloride-strike bath.
(*e*) Overcleaning	Surface of the work should be checked after cleaning for films or stains from an overconcentration cleaner. Excessive current density, especially in anodic cleaning, should be avoided.
(*f*) Undercleaning	Water breaks from a below-concentration cleaner indicate undercleaning. Current density may be too low.
(*g*) Improper acid dipping	Inspection should be made for stains or films from an overconcentration acid dip, or lack of neutralization from an underconcentration acid dip. Muriatic (hydrochloric) acid may be more suitable than sulfuric, or *vice versa*, for the job.
(*h*) Impurities	The plating bath should be tested for impurities such as lead, chromium, or foreign organics. Metallics such as lead are removed by dummy plating. Chromium is removed by a high pH treatment and sometimes with the additional use of potassium permanganate and lead carbonate.* Organics are removed by carbon treatment, either with or without previous oxidation with potassium permanganate, hydrogen peroxide or sodium hypochlorite, as indicated by tests.
(*i*) Passive base metal	Alloys, such as stainless steel, heat-treated steel and nickel alloys, may have to be activated by use of the Wood strike or by electrolytic acid treatment.
(*j*) Porosity of base metal	Thorough rinsing is required to remove entrapped cleaner, acid, etc.

2. DEPOSIT LACKS DUCTILITY

(*a*) pH too low	The pH should be tested and adjusted with nickel carbonate. Ammonia or alkali carbonates should never be used in bright nickels for this adjustment.
(*b*) Excess peroxide	Where hydrogen peroxide is being used as an antipit in dull nickel baths, excesses should be avoided. When an accidental excess occurs, it is removed by heating of the bath. Hydrogen peroxide in excess may cause pitting or may result in a deposit that is passive to chromium plate; also the ductility of the deposit may be impaired.
(*c*) Low boric acid	Boric acid may be very much below normal; analysis will indicate the additions that are required.

* W. A. WESLEY and W. H. PRINE 'Practical Nickel Plating', p. 30. Published by The International Nickel Co., N.Y., 1952.

Possible Source of Trouble	Suggested Cure	Possible Source of Trouble	Suggested Cure
(*d*) Organic contamination	Organic impurities should be checked and removed. The effect of organic impurities should be determined by use of Hull Cell tests. They are removed by carbon treatment, with or without preliminary oxidation, as tests may indicate, and, as detailed in the section on impurities (1*h*).		booster type, being in excess. The amount of excess should be determined and then reduced in concentration to the proper level. Booster brighteners are those that give the greatest brightness, are required more frequently as additions to a bath and normally are present in a small optimum concentration as compared to the other additions.
(*e*) Excess brighteners	Lack of ductility in bright nickel baths results from brighteners, especially		
3. DEPOSIT IS DULL (BRIGHT NICKEL BATHS)			
(*a*) pH out of range	The pH should be adjusted to an optimum value.		are removed by carbon treatment, with or without preliminary oxidation, as tests may indicate and as outlined in the section on impurities (1*h*) above.
(*b*) Booster brightener low	Analysis for this material indicates the addition that is required.		
(*c*) Temperature out of range	The temperature should be kept within range.	(*f*) Metallic impurities	The bath should be analyzed for presence of copper, zinc, lead and cadmium. These impurities are removed by dummy plating, precipitation-filtration or both.
(*d*) Brightener additions out of balance	Where two or more addition agents are used, the proper proportions must be present. Adjustments should be made to the correct values.	(*g*) Drag-in contamination	The bath should be checked for contaminating drag-in from cleaners, acid dips, dirty rinses, etc., by cracked or loose rack coatings, or by improper rinsing of recessed work. The source of trouble should be eliminated.
(*e*) Organic impurities	The bath should be tested with a Hull Cell for the presence of organic impurities from rack coating, tank linings, or buffing compounds. When found, they		
4. DEPOSIT IS DARK			
(*a*) Impurities	Excessive amounts of copper, zinc, lead or cadmium may be present. They are removed by dummy plating.		be analyzed to test for this condition, and it should be adjusted then to proper concentration.
(*b*) Concentrations off	A dull nickel bath may plate dark when used diluted and at higher current densities. The bath should	(*c*) pH too high	A dull nickel bath will plate dark at a high pH where basic nickel salts will plate out. Adjustment of pH to proper level corrects the difficulty.

Possible Source of Trouble	Suggested Cure	Possible Source of Trouble	Suggested Cure
5. STREAKY DEPOSITS			
(*a*) Concentrations not normal	The solution should be tested for low concentration of nickel, chloride and boric acid and adjusted to the correct values.		cure this problem. Horizontal streaks may be due to gelatinous material adhering to surface during the deposition of the coating.
(*b*) Gassing due to dirt in crevices or holes	Adhering buffing compounds and dirt can cause this trouble and should be eliminated.	(*e*) Localized low pH	Acid may be retained in holes or crevices due to improper rinsing. Thorough rinses will help to avoid this condition.
(*c*) Contamination present	Cleaner or cyanide may be retained in holes or crevices through improper rinsing. Steps should be taken to eliminate such a condition if they are found.	(*f*) After additions of brightener	Bath should be thoroughly mixed.
(*d*) Foreign material suspended in bath	Filtration of the bath and an investigation into the source of suspended material with its removal should	(*g*) Hydrogen pitting	Vertical streaks may be caused by hydrogen gassing. Agitation, redesign of racks and other steps may be taken to correct this.
6. SPOTTY DEPOSITS			
(*a*) At bottom of work	The bath should be stirred well or agitated to avoid stratification and non-uniform temperature layers or both.	(*c*) Poor rinsing	Cleaners not thoroughly rinsed off, or not neutralized by acid dip, or silicates precipitated on the work will cause spotty deposits. Thorough rinses are advised.
(*b*) Foreign solid material in bath	Filtration of the bath eliminates this source of contamination. Calcium sulfate is less soluble in a hot bath than in a cold one and may precipitate out to cause roughness.	(*d*) Undissolved boric acid	Heating bath to redissolve boric acid, which crystallizes when bath gets too cold, is suggested.
7. DEPOSIT DARK IN RECESSES			
(*a*) Metallics in bath	The bath should be tested for contamination by copper, zinc, cadmium, and lead. If found, they are removed by dummy plating.		by carbon treatment, as outlined in section (1*h*) above.
(*b*) Organic impurities	Hull Cell tests indicate these impurities. They are removed, if found present,	(*c*) Low current density	Increases of current density as high as possible without burning of the work is advised.

Possible Source of Trouble	Suggested Cure	Possible Source of Trouble	Suggested Cure
8. POOR THROWING POWER AND POOR COVERAGE			
(*a*) Metal content low	Analysis for nickel concentration and increases to the proper level are recommended.		ties. These are removed, if found present, by dummying to eliminate the metals and by carbon treatment to remove organics as outlined in (1*h*) above.
(*b*) Conductivity poor	A check of chloride content and an increase to the proper level is indicated.	(*e*) Loose contacts	A check of all electrical connections to tank, rack, and anodes is advised. Cleanliness to insure full flow of current is a must.
(*c*) Current density low	An increase in current, as high as possible without burning of the work is advised.		
(*d*) Impurities present	The bath should be tested for metallic impurities, copper, zinc, cadmium and lead, and organic impuri-	(*f*) Anodes	An inspection of anodes for number, spacing and area is suggested. Normally 2-3 anodes per foot of anode bar are sufficient.
9. BURNT EDGES OR FROSTY PLATE			
(*a*) Excessive current density	A reduction of current or an increase of agitation and a higher temperature are in order. Work should not be too close to the anodes.	(*e*) Chemical contamination: Presence of aluminum and iron causes frosty deposits at high current densities	A test for chromates or nitrates is advised. Chromium is removed by high pH method* and nitrates are reduced by dummy plating at 50–60 amperes per square foot, at a pH of 2–3 electrometric. Aluminum and iron are removed by high pH treatment.
(*b*) Bath concentrations low	The operator is instructed to analyze and build up the bath to standard values.		
(*c*) Ammonium salts present (high current density baths)	Ammonium salts limit maximum current density and should never be added, as they can only be reduced by drag-out.	(*f*) Organic contamination	Hull Cell tests and removal of the contamination, as (1*h*) above, are in order.
(*d*) Excess brighteners	Excess brighteners may limit maximum current density. Brighteners should be kept within proper limits.	(*g*) Excess iron in bath	Analysis for iron in the solution should be made. It is removed by raising of the pH and followed up with filtration of the solution.
10. PITTING			
(*a*) Antipit agent off concentration	If wetting agent is used, pitting may occur if concentration is too low, but also if too high. The operator is cautioned to avoid excesses if possible.	(*b*) Antipit agent	If hydrogen peroxide is being used in a cold bath, concentration may be too low. If organic antipit is being used, the concentration may be low.

* W. A. WESLEY and W. H. PRINE: 'Practical Nickel Plating', p.30. Publishe d by The International Nickel Co., N.Y., 1952.

Possible Source of Trouble	Suggested Cure
(*c*) Organic contamination	Organic contaminants may cause pitting and are removed with activated carbon as outlined in (1*h*) above.
(*d*) Rough deposit pits	Foreign solid material—such as precipitated iron, dirt and carbon—is removed by filtration.
(*e*) If pH and concentrations are normal and bath is clean	Tests should be made for air being introduced by the filter, or by the addition of cold water while plating is being performed. Also a check for lost work dissolving in the bath is advised.
(*f*) Gassing while plating	Zinc base metal, not completely covered with copper, will dissolve and release hydrogen and contaminate the bath.
(*g*) Low pH	Too low a pH will attack zinc base metal through a thin copper plate. Adjustment to best operating level is urged.
(*h*) Low temperature	Adjustment of temperature to optimum range is necessary.
(*i*) Low boric acid	Analysis for boric acid, and a build up to proper concentration are indicated.
(*j*) Current excessive	Lowered current, or increased agitation, and raised temperature are suggested.
(*k*) When heating up bath	A cold bath, especially if not used for some time, will have absorbed air which will pass off after the bath is heated.
(*l*) After raising pH	Carbon dioxide, produced when the pH is raised with nickel carbonate, is removed by heating of the bath.

NOTE: Hydrogen pits are characterized by a bright pit with a vertical tail. Gas pits from air or carbon dioxide are characterized by very fine pits with no tails. Rough deposits are sometimes confused with pitting.

11. ROUGH DEPOSITS

(*a*) High current density	Adjustment of the current density is required, as excessively high current may cause co-deposition of minute solid particles that are suspended in bath. Each bath has a limiting maximum current density.
(*b*) Low bath concentration	Too low a concentration of nickel, chloride, and boric acid will promote rough deposits. Analysis of the bath and adjustments to proper concentration are in order.
(*c*) Low temperature	Very low temperature, below about 60°F., depending on current density will cause roughness. The bath temperature should be adjusted to a practical value.
(*d*) High pH	A pH above 5·0 electrometric may promote inclusion of basic nickel salts in the deposit, depending on current density. An adjustment of the pH to a proper level is advised.
(*e*) Iron and aluminum contamination	An excess of dissolved iron and aluminum will promote precipitation under electrolysis at the high pH's. The impurities are removed by high pH precipitation plus filtration.
(*f*) Solids in bath	Filtration of the bath is recommended. Operators are cautioned to avoid the presence of anode sludge, dirt, buffing compounds, filter-aids, activated carbon in the solution.

ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Minerals used in the Chemical and Allied Industries: Handbook

S. J. JOHNSTONE: 'Minerals for the Chemical and Allied Industries.' *Published by Chapman and Hall, Ltd., London*, 1954; 692 pp. Price 75/-.

The object of this book is to present, in a concise form, essential information on sources of supply, processing, and metallurgy, together with reference to specifications laid down for the use of the respective materials in individual industries. The subject matter forming the basis of the book appeared in a series of articles published, in the period 1946–49, in *Industrial Chemist*, but new information which has become available in the interim has been integrated, and various sections have been revised in the light of advances which have recently taken place. New chapters have been written on additional minerals and metals which have lately assumed commercial importance. Over 80 materials are included in the survey.

The chapter on *Nickel* contains sections on world production, metallurgical treatment of nickel-containing ores, refining processes, specifications for various grades of nickel, uses of nickel-containing steels and non-ferrous alloys, uses of nickel salts and compounds, electroplating, nickel-alkaline accumulators, and other applications of nickel and nickel-containing materials. Some figures for consumption of nickel in U.K. and U.S.A. are also included.

Specification for Metallic Electrical-Resistance Materials

BRIT. STANDARDS INSTN.: 'Metallic Resistance Materials for Electrical Purposes.' *B.S. 115–1954*; 13 pp. (revised edition of 1938 specification).

The Standard specifies the electrical and mechanical properties of metallic resistance materials in the form of wire, strip, tape, and sheet. The materials are divided into three main classes, based on temperature coefficient of resistance and working temperature.

An appendix contains details of the sizes of wire in general use.

Effects of Nuclear Radiation on Constructional Materials

D. O. LEESER: 'How Nuclear Radiation Affects Engineering Materials.' *Materials and Methods Manual No.* 107: *Materials and Methods*, 1954, vol. 40, Aug., pp. 109–20.

Engineering materials used in the construction of atomic-power reactors are not only subject to heat and to highly corrosive conditions, but are also exposed to bombardment by nuclear particles. Information on the effects of such bombardment, on the properties and behaviour of metallic and non-metallic materials, is gradually being accumulated, and this manual presents a preliminary survey of data already assembled.

It is emphasized that the exact nature of the changes due to radiation damage is not yet fully understood. In the case of metals and alloys the effect of radiation had been presumed to be broadly similar to that of cold-work, with the exception that, in the case of radiation the effect is on a microscopic or extremely localized scale and is non-directional. Recent investigations have, however, led to some suspicion as to the validity of this presumption, and there is now a tendency to explain the mechanism of radiation as not corresponding directly to cold-work but as being somewhat more closely analogous to solid-solution hardening. The theory is diagrammatically illustrated in this article.

Although there is thus some uncertainty as to the mechanism of the effect produced, there is clear evidence that changes in the properties of materials do occur as the result of exposure to radiation, and it is obvious that consideration must be given to such changes in selecting materials for use in atomic power plants.

This manual comprises a clearly presented summary of the effects of radiation on hardness, tensile strength, impact strength, fatigue strength, density and dimensional stability, magnetic susceptibility, and electrical resistivity, of a range of steels and non-ferrous materials. Materials for which data are tabulated, in one or more of these sections, include carbon steels, stainless (high-chromium and austenitic chromium-nickel) steels, nickel, Hastelloy (nickel-molybdenum-base alloys), Monel and K Monel (nickel-copper-base alloys), Inconel and Inconel X (nickel-chromium-iron-base alloys), and cobalt-base alloys. A summary is also made of information already accumulated on the effects of radiation on non-metallic materials. Diagrams show types of test specimen which have been used for study of radiation damage, and a note is added on test methods employed.

See also—

C. R. SUTTON and D. O. KEESER: 'How Radiation Affects Structural Materials.' *Iron Age*, 1954, vol. 174, Aug.19, pp. 128–31; Aug. 26, pp. 97–100.

This article, containing information closely similar to that embodied in the Materials and Methods Manual, is based on a paper presented by the authors at a Congress on Nuclear Engineering, recently sponsored by the American Institute of Chemical Engineers.

Electrographic Identification of Steels and Alloys

E. FITZER: 'Rapid Electrographic Identification of Steels and Alloys.' *Archiv. f.d. Eisenhüttenwesen*, 1954, vol. 25, July.-Aug., pp. 321–6.

The increasing complexity and variety of metallic materials now in commercial use has intensified the need for rapid, non-destructive methods by which rapid identification, and if possible roughly quantitative determination of composition, can be effected. The author briefly reviews the methods which have been proposed to meet this need, indicating their scope of usefulness and their limitations.

The basic principles of electrography are then summarized, and reference is made to the extent to which these principles have been usefully developed for identification of metals and alloys.

The main portion of the paper comprises a series of identification procedures, using drop-test reactions adapted for electrographic operation. Methods are given for identification and approximate quantitative determination, (in ferrous and some non-ferrous materials), of chromium, cobalt, nickel, manganese, molybdenum, tungsten, titanium, silicon, and copper. Procedures based on these reactions have been devised for general identification of alloys of various classes. The recommendations made are illustrated by charts showing the successive steps involved.

The author is of the opinion that full development of electrographic testing has been hindered by the lack of simple, robust equipment suitable for use under routine conditions. He describes, with illustrations, two improved types of apparatus with which the recommended methods may be carried out.

Directory of British Hardfacing Electrodes

'Directory of Welding and Fabricating Equipment. Part 3. Hardfacing Electrodes.'
Welding and Metal Fabrication, 1954, vol. 22, July, pp. 280–4; Aug., pp. 319–22.

The directory gives the trade names of hardfacing electrodes manufactured by the various British producers, sizes of electrode available, characteristics of the deposits made by them, typical applications, etc.

Deposition of Metal Coatings by Evaporation

See abstract on p. 191

NICKEL

Minerals used in the Chemical and Allied Industries: Handbook

See abstract on p. 188.

Stress in Evaporated Nickel Films

R. W. HOFFMAN, R. D. DANIELS and E. C. CRITTENDEN: 'The Causes of Stress in Evaporated Metal Films.' *Proc. Physical Soc.*, Sect. B, 1954, vol. 67, June 1, pp. 497–500.

Metal films condensed from vapour in high vacuum are found to be in a state of relatively high tensile stress, and a mechanism for the origin of the stress has recently been proposed by MURBACH and WILMAN (*ibid.*, 1953, vol. 66, p. 911). Their interpretation is that the films are relaxed to a stress-free condition at roughly the recrystallization temperature, during deposition, and that subsequent cooling to the temperature of the substrate gives rise to stress as a result of thermal expansion. The present note suggests an alternative mechanism, based on observations made with films of nickel and other metals deposited from the vapour, on substrates at various temperatures. Measurements of the stress in the films were made with the substrate at 25°C.

According to the theory proposed, the intrinsic stress present in a film under given conditions is believed to be the result of the history of its imperfections. These include the crystal boundaries, as well as imperfections within the crystals which make up the polycrystalline film. Imperfections which are formed during deposition undergo change shortly after being trapped by succeeding layers of deposited metal: these changes take place at essentially the temperature of the substrate, influenced perhaps to some extent by the temperature pulses from atoms arriving on the surface.

The authors consider it likely that the irregular regions on the surface being built up, which become crystal boundaries when buried by continuing deposition, rearrange, and give rise, through a reduction in volume, to the principal part of the intrinsic stress. Total stress usually includes a component resulting from differential expansion relative to the substrate; this is normally small compared with the intrinsic stress.

Thickness/Coercivity Relationships in Nickel and Nickel-Iron Alloys

V. A. ZAIKOVA and Y. S. SHUR: Dependence of the Coercivity of Magnetically Soft Materials on the Thickness of the Lamination.' *Comptes Rendus, Acad. Sci., U.R.S.S.*, 1954, vol. 94, Feb. 1, pp. 663–5:
Wireless Engineer, 1954, vol. 31, Aug., Abs. 2444.

'Experimental investigation of the coercivity of laminations of iron, nickel, iron-nickel alloys containing 36–87 per cent. of nickel, and iron-silicon alloys containing 1–4 per cent. of silicon. In all cases but one a critical thickness between 0·03 and 0·07 mm. was found, below which coercivity increases very rapidly. The results are shown graphically and are tabulated.'

Promotion of Nickel Catalysts

R. B. BLANCE and D. T. GIBSON: 'The Promotion of Raney Nickel Catalysts.' *Jnl. Chemical Soc.*, 1954, July, pp. 2487–9.

In addition to continuous improvements in the methods of preparation of Raney nickel catalysts, it has been found that, for many reductions, the catalyst is promoted by treatment with various addition reagents. Delépine showed that, in the hydrogenation of ketones, Raney nickel is promoted by treatment with chloroplatinic acid followed by sodium

hydroxide: Lieber found that treatment with triethylamine and chloroplatinic acid is also effective. The present authors demonstrate that a combination of the Delépine and Lieber techniques gives better results than can be obtained by either used alone: they also show that lithium hydroxide can with advantage replace sodium hydroxide.

A. V. BUKHMAN and D. V. SOKOL'SKII: 'Hydrogenation of Linseed Oil on Nickel Catalyst on Silica Gel Promoted by Platinum and Palladium.'
Izvest. Akad. Nauk Kazakh. S.S.R., 1953, No. 123, Ser. Khim., No. 7, pp. 9–19.

N. I. SHCHEGLOV and D. V. SOKOL'SKII: 'Hydrogenation of Cottonseed Oil in the Presence of Raney Nickel Catalyst Promoted by Platinum and Palladium.' *ibid.*, pp. 30–8.

Abstracts of both papers appear in *Chemical Abstracts*, 1954, vol. 48, May 25, p. 6145.

Ferromagnetic Resonance in Colloidal Nickel

D. S. M. BAGGULEY and N. J. HARRICK: 'The Temperature Dependence of Ferro-Magnetic Resonance in Colloidal Nickel.' *Proc. Physical Soc.*, Sect. A, 1954, vol. 67, July 1, pp. 648–9.

Measurements of ferro-magnetic resonance in colloidal suspensions, made by the senior author in 1953, have been extended to investigation of the temperature-dependence of the line width and spectroscopic splitting factor (g) for nickel between 20° and 750°K. (−253° and +477°C.). Measurements made on nickel foil by BLOEMBERGEN (*Physical Rev.*, 1950, vol. 78, p. 572) led to the conclusion that the g value was constant between room temperature and the Curie point (631°K.: 358°C.). The studies now reported give a more accurate confirmation of this conclusion, and show, in addition, that a discontinuity occurs at the Curie point, where the g value falls from $2{\cdot}22\pm 0{\cdot}02$ to $2{\cdot}00\pm 0{\cdot}05$. This considerable change at the Curie point was not to be anticipated, and is not readily explained by current theories of ferromagnetism. The measured line width has an anomaly near the Curie point, where it increases rapidly from a minimum value of 350 gauss ±10 per cent. to about 850 gauss ±30 per cent. in the paramagnetic region.

Solubility of Nickel Dimethylglyoximate

H. CHRISTOPHERSON and E. B. SANDELL: 'Molecular and Ionic Solubility of Nickel Dimethylglyoximate.'
Analytica Chimica Acta, 1954, vol. 10, pp. 1–9.
Abstract in *Chemical Abstracts*, 1954, vol. 48, May 25, p. 5723.

Determination of Nickel in Cobalt

E. M. GOLDSTEIN: 'Determination of Nickel in Cobalt or its Compounds.' *Chemist Analyst*, 1954, vol. 43, June, pp. 42-3.

Dimethylglyoxime is not suited to the estimation of small amounts of nickel in the presence of larger amounts of cobalt because a solution of cobalt dimethylglyoximate perceptibly dissolves the corresponding nickel complex. If, however, the relative concentration of the cobalt can be reduced, nickel can be determined with dimethylglyoxime.

SCHOELLER (*Analyst*, 1944, vol. 69, p. 8) separated cobalt from nickel by boiling an ammoniacal solution of their phosphates: cobalt phosphate is slowly precipitated as cobalt ammonium phosphate, whilst nickel remains in solution. Precipitation of cobalt by this method is, however, incomplete, because cobalt (II) is oxidized to cobalt (III) during boiling, and soluble pink ammonia complexes are formed. It has now been found that this oxidation can be substantially suppressed by addition of a small quantity of a reducing agent such as hydroxylamine. The following procedure is recommended:—

'*Reagent*. Dissolve 20 g. disodium phosphate, 25 g. ammonium chloride and 2·5 g. hydroxylamine hydrochloride in 800 ml. of water, and add 150 ml. of concentrated aqueous ammonia.

'*Procedure*. Take an aliquot of a solution of the metal or the cobalt salt corresponding to about one gram of the metal and add it to about 200 ml. of the reducing ammoniacal phosphate solution. If necessary, add concentrated aqueous ammonia to dissolve as much as possible the precipitate which first forms. Boil the solution for about 5 minutes, taking care to prevent losses by bumping. Cool the solution somewhat; filter off the precipitated cobalt compound and wash with water containing a little ammonia. The clear filtrate contains all of the nickel, with only a trace of cobalt, and it is suitable for the precipitation of nickel by dimethylglyoxime.'

The procedure described makes possible easy and accurate determination of as little as 0·02 per cent. of nickel in cobalt metal or cobalt compounds.

Electrographic Identification of Steels and Alloys

See abstract on p. 189.

Basic Nickel Carbonates

J. T. CARRIEL and W. J. SINGLEY: 'Composition of Basic Nickel Carbonates.' *Jnl. Amer. Chemical Soc.*, 1954, vol. 76, July 20, pp. 3839–43.

Basic nickel carbonates are frequently used as catalyst intermediates, and it has been observed that minor changes in mode of preparation may in some cases influence the behaviour of the final reduced-nickel catalyst. This paper is concerned with establishment of fundamental data which could assist elucidation of problems associated with preparation of catalysts: specifically, the object of the investigation was to determine the influence of pH of the reaction medium, on the composition of the basic nickel carbonates formed.

Basic nickel carbonates were formed at a constant pH and room temperature, by addition of standard nickel chloride and potassium hydroxide solutions to a reaction medium containing a 10-fold excess of potassium carbonates. The composition of the precipitates may be expressed as $Ni(OH)_2.3$ to $4NiCO_3$ in the pH range 8 to 10·5: above pH 10·5 the ratio

of carbonate to hydroxide in the precipitate decreases rapidly. Essentially no chloride is absorbed by the precipitate. A method of precipitation at constant pH, previously developed by the authors, was adapted to permit calculation of the composition of the precipitate directly from burette readings.

Effects of Nuclear Radiation on Constructional Materials

See abstract on p. 188.

ELECTRODEPOSITION AND OTHER COATING METHODS

Nickel-Dip Treatment in Enamelling of Sheet Steel

D. G. MOORE, J. W. PITTS and W. N. HARRISON: 'Rôle of Nickel in Enamelling of Sheet Steel.' *Nat. Advisory Committee for Aeronautics, Tech. Note* 3207, June, 1954; 27 pp.

Nickel-dip, or nickel-flash, treatment is extensively used in the porcelain-enamelling industry, to improve the quality of the coatings. The process consists of immersing the work for about 5 minutes in a weak aqueous solution of nickel salts, at 150°–170°F. (65°–75°C.), and at a pH adjusted to about 3·0. During this treatment a thin film of metallic nickel is plated onto the steel by a galvanic replacement reaction, which can be represented as follows: $NiSO_4 + Fe \rightarrow Ni + FeSO_4$.

The presence of the nickel film on the steel is claimed (1) to ensure adherence of the coating to the metal over a wider range of time and temperature in the firing process than is obtainable with unflashed steel, and (2) to reduce incidence of fishscaling and copperheading.

Various theories have been advanced to explain the improved adherence resulting from the nickel-dip treatment: one of these postulates that the thin layer of nickel retards oxidation of the steel during firing and that since too much oxide at the interface is detrimental to adherence, nickel improves the bond: the other theory is that good adherence results from roughening imposed on the steel surface by a molten enamel containing either cobalt or nickel ions. According to this theory adherence is caused mainly by mechanical gripping. If, however, the nickel dip produces adherence by a mechanism other than a surface-roughening action, an understanding of the mechanism responsible is essential to complete elucidation of the phenomenon of ceramic-to-metal adherence.

The aim of the work described in the present paper was therefore two-fold:—(1) quantitative determination of the effects of the nickel dip on development of adherence and on prevention of fishscaling, and (2) study of the mechanism by which nickel aids in producing an adherent bond. The research comprises one phase of a broad study on adherence of ceramics to metals, which is being conducted at the National Bureau of Standards, under the sponsorship of the National Advisory Committee for Aeronautics.

Two basis materials were used; enamelling iron, and titanium-stabilized low-carbon steel. Full compositions of both are given, together with details of the frits employed, and of preparation of the specimens. Measurement of adherence was by means of a tentative standard test developed by the Porcelain Enamel Institute, in which the strength of the bond is evaluated in terms of the amount of metal exposed after standard deformation treatment, expressed as a percentage of total deformed area.

From the results of the tests, which are reported in a series of tables and figures, the following conclusions are drawn, as being justified in the case of a single-frit ground-coat enamel, both with and without cobalt oxides:—

'The nickel dip produced a large improvement in adherence index with specimens fired for short times, but the effect of the nickel on adherence decreased markedly as the firing time was increased.

'Under most conditions, the nickel dip produced a larger increase in adherence index in the case of a titanium-bearing low-carbon steel than in the case of enamelling iron.

'An optimum weight of nickel deposit was found for the development of maximum adherence index for the titanium-bearing steel. This optimum varied from 50 to about 120 milligrams per square foot, depending on the type of cleaning used and the type of enamel applied. Similar determinations were not made in the case of the enamelling iron.

'The nickel dip had a beneficial effect in reducing fishscaling.

'Nickel dipping of the steel prior to enamelling brought about a surface roughening during the firing operation. A relationship was noted between the degree of roughening and the measured adherence index.

'The function of the nickel dip in improving adherence is believed to be associated with galvanic attack. The nickel apparently deposits from the aqueous solution selectively on more cathodic areas of the steel surface, thus forming local galvanic cells which begin to function immediately after the enamel fuses and wets the metal surface. During firing the iron members of the cells corrode, while the nickel areas are cathodically protected. The result is that the surface becomes roughened and the coating appears to anchor itself to the undercut surface.'

Wear-Corrosion Tests on Metal Coatings in High-Temperature Water

See abstract on p. 202.

Deposition of Nickel Coatings by Evaporation

W. L. BOND: 'Notes on Solution of Problems in Odd Job Vapor Coating.' *Jnl. Optical Soc. of America*, 1954, vol. 44, June, pp. 429–38.

The author gives a series of charts showing conditions necessary for the production of high-quality metal coatings of given optical transmission and given electrical resistance, as well as specified thicknesses or weight per unit area. Nickel coatings are among the wide range considered. Problems arising in deposition by this method are discussed in considerable detail.

NON-FERROUS ALLOYS

Minerals used in the Chemical and Allied Industries: Handbook

See abstract on p. 188.

Monel Filters in Paper Mills

H. A. RONNINGEN: 'How Iron Bacteria are being Successfully Removed with Wire Cloth Filters.' *Paper Trade Jnl.*, 1954, vol. 138, June 18, pp. 26, 28.

In some paper mills using deep-well water serious plugging of shower nozzles, sprays, valves and other parts occurs, due to the high iron content of the water. A reddish brown ferric-oxide slime is formed, which, in addition to causing clogging, results in losses in bleaching, and produces stains. Water having an iron content of over 0·5 p.p.m. is particularly troublesome.

This article describes fine wire-mesh filters which are being successfully used in parts of the U.S.A. to obviate these difficulties. The filter cloth is of Monel (nickel-copper alloy), chosen because the material is tough and strong, and therefore stands up well to the severe conditions imposed, and because Monel can, without damage, be 'soured out' with 10 per cent. sulphuric or 10 per cent. muriatic acid solution.

Specification for Metallic Electrical-Resistance Materials

See abstract on p. 188.

Effects of Nuclear Radiation on Constructional Materials

See abstract on p. 188.

Determination of Chromium in Alloy Steels and Copper-base Alloys

See abstract on p. 196.

Electrographic Identification of Steels and Alloys

See abstract on p. 189.

NICKEL-IRON ALLOYS

Structure of Iron-Nickel-Aluminium Permanent-Magnet Alloys

E. A. NESBITT, H. J. WILLIAMS and R. M. BOZORTH: 'Factors Determining the Permanent Magnet Properties of Single Crystals of Fe_2NiAl.' *Jnl. Applied Physics*, 1954, vol. 25, Aug., 1014–20.

It is shown in this paper that fine precipitated particles present in single crystals of Fe_2NiAl are elongated, and that the shape anisotropy of these particles is a major factor in determining the coercive force of the alloy. For fields of less than 7,000 oersteds ⟨111⟩ directions are those of easy magnetization in the (110) plane of the crystal. This is due to the effect of the shape of the precipitated particles, which are oriented in three mutually perpendicular directions. In higher fields the torque due to this effect decreases and the [001] direction is then the one of easy magnetization, because of intrinsic crystal anisotropy. These conclusions are based on an analysis, and on measurements, of the torque on three mutually perpendicular rods of magnetic material, which simulated the precipitated particles in the actual crystal.

Anhysteretic Magnetization in Nickel-Aluminium-Iron-base Magnet Alloy

J. E. GOULD and M. McCAIG: 'Anhysteretic Magnetization of Alcomax III.' *Proc. Physical Soc.*, Sect. B., 1954, vol. 67, July 1, pp. 584–6.

Curves illustrate anhysteretic and normal magnetization in the preferred and transverse directions in various fields: practical consequences of the observations made are discussed. The measurements recorded do not confirm suggestions which have been made as to the possible presence, in alloys cooled in a magnetic field, of oriented submicroscopic precipitated plates.

Thickness/Coercivity Relationships in Nickel and Nickel-Iron Alloys

See abstract on p. 189.

Nickel-Iron Alloys in Magnetic Amplifiers

G. M. ETTINGER: 'Magnetic Amplifiers. A General Survey.' *Electrical Rev.*, 1954, vol. 155, Sept. 3, pp. 348–52.

Design, mode of operation, and materials of construction are dealt with in this comprehensive article. *Inter alia*, composition, magnetic properties and trade names are tabulated for nickel-iron-base alloys of types recently developed for use as magnetic core materials.

Electrographic Identification of Steels and Alloys

See abstract on p. 189.

Nickel-Iron Alloys in Relay Cores

J. P. MARTIN: 'Design Requirements of Relay Core Materials.' *Electrical Manufacturing*, 1954, vol. 53, Apr., pp. 138–40.

There are six main requirements for core materials used in high-quality relays:—high induction, high permeability, low residual magnetism, low coercive force, non-ageing characteristics, good physical properties. In this article the reasons for such requirements are briefly outlined, as an introduction to a detailed discussion of the influence of design on operating efficiency of relays. It is pointed out that the crux of the relationship of desired magnetic characteristics of relay core materials to design factors lies in the air-gap ratio: the effects of air gaps and of pole shape are dealt with at some length. A table shows the magnetic properties of core materials suitable for use in relays: these include several types of nickel-iron alloy.

CONSTRUCTIONAL STEELS

Ultra-High-Strength Steels for Aircraft

SOC. AUTOMOTIVE ENGINEERS: 'Symposium on Ultra-High-Strength Steels in Aircraft Applications.' S.A.E. *Special Publn.* 120, 1953; 73 pp.

The scope of the individual papers presented at this symposium, held in March, 1953, is shown below:—

C. WELLS: 'Composition and Heat-Treatment of High-Strength Steels', pp. 1-24.

The paper is restricted mainly to presentation and interpretation of data on transverse mechanical properties. It is urged that transverse reduction of area and keyhole or V-notch Charpy impact values are better indications of the quality of steel, with regard to ductility and toughness, than are comparable longitudinal test values. Tabular and graphical data are presented to demonstrate the influence of composition on mechanical strength, ductility, toughness, hardenability; the influence of section size; the liability of various types of steel to quench-cracking; the influence of slack quenching, double tempering and triple tempering, on transverse ductility; the effect of low-temperature tempering and of strain-ageing, on longitudinal toughness; the effect of grain direction on mechanical properties.

The conclusions drawn from the data recorded (which are summarized below), although based in many cases on values for quenched-and-tempered steels having tensile strengths below 200,000 p.s.i. (90 tons per sq. in.), are believed to be generally applicable to steels of higher tensile strength, and are considered to provide a sound basis for development of ultra-high-strength steels:—

Increase in carbon raised the tensile strength of quenched steels subjected to a given tempering temperature. The lower this temperature, the higher the tensile strength and the larger the increase in strength due to added carbon. Carbon, molybdenum, chromium, manganese, silicon, and vanadium each raised the tensile strength of fully quenched and tempered steels; boron and nickel had no appreciable influence.

Steels of various compositions, fully quenched, tempered at sufficiently high temperatures, and having sufficiently high tensile strengths, all showed a yield strength/tensile strength ratio close to 0·94. For steels tempered in the range 500°–800°F. (260°–425°C.) the ratio increased with rise of tempering temperature.

Added carbon and silicon lowered transverse ductility; molybdenum and chromium slightly raised it; nickel and vanadium had practically no effect.

In steels fully quenched, and tempered to high tensile-strength levels, added carbon lowered transverse toughness, whereas nickel, chromium, silicon, molybdenum and vanadium had practically no effect. Manganese lowered longitudinal toughness.

Additions of alloying elements or intensifiers containing boron, to increase inadequate hardenability, improved strength, ductility and toughness. In a slack-quenched steel, nickel, as would be anticipated, improved transverse toughness; the influence of boron, on slack-quenched steels, varied according to the basis composition.

The presence of 10 per cent. of bainite, a product of slack quenching, did not significantly affect either transverse ductility or transverse toughness of specimens consisting of isothermal bainite and martensite tempered to a high tensile strength. In a large quenched-and-tempered forging the products of slack quenching lowered transverse ductility much more severely at high than at low tensile-strength levels.

Susceptibility to quench cracking increased with rising carbon content and decreased with an increase in the degree of slack quenching.

Optimum tempering temperature for obtaining maximum longitudinal toughness at tensile strengths above 200,000 p.s.i. (90 tons per sq. in.) in good-quality nickel-chromium-molybdenum steels of the 'Krupp'* type containing 0·26 per cent. of carbon was found to be 400°F. (205°C.). The blue-brittle range of this steel was observed to be high.

Strain-ageing treatment applied to a number of quenched martensitic low-carbon steels increased tensile strength (with some sacrifice of ductility) and raised yield strength/tensile strength ratio as desired, without appreciably altering the tensile strength or decreasing either ductility or toughness.

It is observed that strength properties are isotropic, whereas ductility and toughness are anisotropic. Difference between average transverse and longitudinal reduction of area increases with rising tensile strength.

J. M. HODGE, R. D. MANNING and J. A. BAUSCHER: 'The Development of Steels for Use at High Strength Levels', pp. 25-9.

This paper summarizes results of research carried out by the United States Steel Corporation.

An evaluation was made of the properties of steels over a wide range of composition. Some contained boron, others were boron-free; in some the silicon was higher than normal; some had high, and some relatively low, manganese contents; in others the nickel was varied from low to comparatively high percentages. In some cases the steels were prepared in the laboratory induction furnace, in others they were made in a 7-ton electric furnace. Detailed analyses of all types are given in the paper.

Tests made included end-quench hardenability determinations; impact tests at normal and at sub-zero temperatures, and evaluation of transition temperatures; assessment of fatigue strength, and ratio of endurance limit to tensile strength.

The results of these tests established the soundness of the basic premises which had been made in starting the investigation, viz., that the essential factors in obtaining steels combining toughness with high hardness would be:—

(1) hardenability sufficient to ensure full tempered martensitic microstructures in the quenched-and-tempered sections,

(2) the use of as low a carbon content as is consistent with the desired level of tensile strength,

* nickel 4, chromium 1·5, per cent., approx.

C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %	B %
0·24	0·53	0·80	0·028	0·017	2·24	0·73	0·53	—
0·35	0·71	0·92	0·025	0·014	0·88	0·83	0·54	0·0039

(3) avoidance of tempering in a temperature range which may lead to embrittlement.

The properties shown by the respective types of steel tested led to intensification of work on two compositions, of which details are shown in the Table above.

Detailed information is given on the properties of these steels, tempered at various temperatures after quenching. The data indicate that both exhibit the desired high degree of toughness, at tensile-strength levels of 220,000-260,000 p.s.i. (98–116 tons per sq. in.), and that they are adaptable to production in large ingots to meet aircraft-quality standards. Both are recommended for use in applications in which a combination of high strength and toughness is essential.

J. W. SANDS: 'Development of a Steel for the 280,000 to 300,000 p.s.i. Tensile Strength Bracket', pp. 35–46.

The author reports research carried out by International Nickel Company, Inc.

Theoretical considerations derived from existing knowledge on the properties of ultra-high-tensile steels already developed indicated the desirability of minimum feasible carbon content, the use of the S.A.E. 4340* type of composition as a basis for modification, and increase in silicon content as a means of retarding softening on tempering. On this basis the following composition was selected as a starting point:—

carbon 0·40, silicon 1·60, manganese 0·70, nickel 1·90, chromium 0·90, molybdenum 0·45, vanadium 0·07, per cent.

Study of the influence of the individual elements contained in this steel, and comparison of the properties of the original steel and of various modified types (including nickel-free varieties), was made by means of hardenability rating, and by determination of mechanical properties and impact-resistance, ductility, and susceptibility to embrittlement. As a result of these tests, the composition in the above Table is recommended as providing steels which offer a desirable combination of hardenability and mechanical properties at tensile strengths approaching 300,000 p.s.i. (134 tons per sq. in.), and yield strengths of the order of 250,000 p.s.i. (112 tons per sq. in.).

* Carbon 0·38–0·43, nickel 1·65–2·00, chromium 0·70–0·90, molybdenum 0·20–0·30, per cent.

The following properties are quoted as typical of this steel:—

	p.s.i.	t.s.i.
Tensile Strength	294,000	131
Yield Strength (0·2% offset) ..	247,500	111·5
Elongation, % in 2 in.	10	
Reduction of Area, %	34	
Impact: Charpy V, at r.t., ft.-lb. ..	16·5	
„ at −50°F., ft.-lb.	15	
Hardness, Rockwell C	54	

Recommended heat-treatment is normalizing at 1700°F. (925°C.), oil-quenching from 1600°F. (870°C.) and tempering at 600°F. (315°C.).

C. E. MOELLER: 'Processing of High-Strength Steel Parts', pp. 47–54.

The experience recorded in this paper is based on the processing of landing-gear components made from S.A.E. 4340 nickel-chromium-molybdenum steel heat-treated to a tensile strength of 260,000-280,000 p.s.i. (116–125 tons per sq. in.). Controlled heat-treatment, of which details are given, is employed to produce a structure ensuring machinability and satisfactory behaviour in other processing operations.

G. G. WALD: 'Processing of Highly Heat-Treated Steel', pp. 55–64.

Lockheed Aircraft Corporation has, over a period of some years, carried out a comprehensive investigation designed to evaluate the properties of nickel-chromium-molybdenum steel of the S.A.E. 4340 composition heat-treated to levels of mechanical strength considerably above those conventionally used in aircraft construction. The results have shown that this type of steel, in the 260,000–280,000 p.s.i.

C %	Si %	Mn %	Ni %	Cr %	Mo %	V %	Al %
0·38–0·43	1·50–1·70	0·60–0·90	1·8–2·0	0·70–0·95	0·30–0·50	0–0·10	0·08
Nominal composition—							
0·40	1·60	0·70	1·90	0·90	0·45	0·07	0·08

condition, is well suited for such applications and that production processing of the steel in that condition presents no serious problems.

In this paper processing and fabrication are discussed with reference to (*a*) their effects on the structural characteristics of the steel, (*b*) their influence on uniformity of the finished product, (*c*) the retention of maximum toughness, and (*d*) the minimizing of

residual stresses in the fabricated parts. On the basis of many test results, the following recommendations are made, with arguments supporting the conclusions drawn:—

All parts should be normalized prior to heat-treatment. The normalizing temperature is critical, and the time required is longer than soaking times usually employed.

The temperature of austenizing or heat-treatment should be kept as low as possible, in order to minimize the amount of retained austenite. Tempering should be done at 400°F. (205°C.) or a higher temperature, and the duration of tempering should be longer than that normally used: the exact temperature should be determined from hardenability data or by measurement of the quench hardness of the actual part.

Decarburization must be avoided: even mild decarburization appreciably reduces fatigue strength at the higher tensile levels.

Retained austenite present after heat-treatment should be stabilized: ageing for 24 hours at 250°F. (120°C.) is suitable.

Hardness/tensile strength relationships must be based on determinations on the nickel-chromium-molybdenum steel in the ultra-high-tensile condition: published data on relationships obtaining for other steels and for 4340 in other conditions are not applicable to the steel when heat-treated to the very high tensile level.

On account of the added difficulty in straightening steel in this condition, particular care should be taken to avoid distortion, and, where parts must be straightened, subsequent stress-relief treatment and peening must be applied to relieve the high residual tensile stresses.

Flash-welding of the high-tensile steel requires exact control: conditions must be pre-standardized and frequently checked.

Machinability, in the majority of cases, is excellent. By the use of special techniques, steel parts in the ultra-high-tensile condition can often be machined more rapidly than steels of lower tensile range machined by normal methods. Tool wear is, however, considerably greater. Grinding is to be avoided if possible, in order to prevent cracking: where such treatment is inevitable it should be followed by light shot-peening.

Following the investigation of mechanical processing, a detailed study was made of cleaning and of pre-plating procedures, with particular reference to the problem of hydrogen embrittlement. Methods for relief of embrittlement were also considered. Full details are given of the experimental work, from which the following conclusions are drawn:—

Cathodic cleaning prior to plating should be prohibited.

Chromium plating should be eliminated from radii.

Ground parts and any other parts containing high residual stresses should be shot-peened prior to plating.

Plated parts should be baked for 8 hours at 375°F. (190°C.), instead of the 4-hour treatment required for plated steels of lower tensile strength.

By conforming with these recommendations adequate relief is obtained from embrittlement likely to result from normal processing operations.

D. A. REDWINE: 'Design Considerations in the Use of Ultra-High-Strength Alloy Steels in Aircraft', pp. 65–73.

Design objectives, and the means of attaining them, are discussed from the point of view of the requirements of the prospective customer, who may be presumed to insist on excellent performance, safety, reliability, minimum maintenance, and adequate operational life.

Some possible causes of failure are considered in relation to the influence which such occurrences should have on approach to design. In connexion with assessment of load-carrying ability, a description is given of the walking-beam sustained-load test used by Vultee Aircraft Corporation: the influence of (*a*) heat-treatment, and (*b*) plating, on behaviour under loading, is critically assessed.

Overall experience leads the author to recommend that prior to use in design, ultra-high-tensile steels should be compared exhaustively with steels which have given consistently satisfactory performance over long periods: that the presence of internal stresses, and means for their relief, be considered as a matter of primary importance; that the use of chromium plating be reduced to a minimum, and that design of joints be treated as a particularly vital factor in aircraft components.

U.S. Boron-containing Nickel-Alloy Steels

'A.I.S.I. Standard Boron Steel Compositions.' *Metal Progress*, 1954, vol. 66, Aug., p. 112B.

Full details of composition limits of boron-containing openhearth and electric-furnace alloy steels, revised as of February, 1954. Steels containing low percentages of nickel, in association with chromium and/or molybdenum, are included in the lists.

Transformation Temperature of Nickel-Alloy Steels in Relation to Weldability

C. L. M. COTTRELL: 'Transformation Temperature of Alloy Steels Related to Weldability with Low-Hydrogen Electrodes.' *Brit. Welding Jnl.*, 1954, vol. 1, Sept., pp. 409–12.

An earlier paper by the same author reported experiments in which the weldability of a series of low-alloy manganese - nickel - chromium - molybdenum steels, arc-welded with a rutile-coated electrode, was related to the temperature of completion of the austenite transformation in the hard zone during cooling (*Jnl. Iron and Steel Inst.*, 1953, vol. 174, pp. 17–24: *Nickel Bulletin*, 1953, vol. 26, No. 7, pp. 116–17).

In view of the fact that use of low-hydrogen electrodes makes possible welding of a given steel under more severe conditions, tests with low-hydrogen ferritic electrodes were made on the same series of manganese-nickel-chromium-molybdenum steels. In the present paper the liability of the steels to hard-zone cracking, when welded with such electrodes, is

related to the temperature at completion of austenite transformation during cooling. The rapid-action dilatometer employed in the tests was described earlier (*Jnl. Iron and Steel Inst.*, 1954, vol. 176, pp. 273–82).

The results show that when transformation is completed at a temperature above 245°C. no hard-zone cracks are formed, but that when it continues below that temperature cracks occur. The exact position of the critical temperature may vary according to the precise hydrogen content of the low-hydrogen electrodes.

Assessment of the effect of alloying elements on weldability and strength of the plate (expressed as weldability/proof-stress values) gave the following decreasing order of efficiency: chromium, manganese, nickel, carbon.

Determination of Chromium in Alloy Steels and Copper-base Alloys

M. D. KAHN and F. J. MOYER: 'Rapid Photometric Determination of Chromium in Alloy Steels and Bronzes.' *Analytical Chemistry*, 1954, vol. 26, Aug., pp. 1371–3.

The method described is based on treatment of a nitric-hydrochloric acid solution of the sample with perchloric acid, to oxidize the chromium present. The solution is subsequently treated with zinc, followed by addition of hydrogen peroxide and of phosphoric acid. The orange-red colour formed is estimated photometrically.

It is claimed that the procedure exhibits a satisfactory degree of reproducibility and accuracy and that it is simple in operation. It is applicable to a wide range of chromium-containing materials, both ferrous and non-ferrous. Details are given for analysis of both types, and the influence of nickel (which increases absorbance of the solution) is discussed, with calibration curves for nickel-free and nickel-containing materials.

Effects of Nuclear Radiation on Constructional Materials

See abstract on p. 188.

Nickel-Alloy Steel Chain

H. E. McCARTHY and H. F. REID: 'Know Your Chain.' *Reprint from Steel*, Feb. 1, 1954; 4 pp.
Issued by International Nickel Co., Inc.

Working properties are compared for chains of wrought iron, carbon steels of various types, and nickel-alloy steels, indicating the superiority of the alloy types.

Selection of nickel-alloy steels for this application is dictated in part by the need for a composition in which strong welds can be produced on an economical mass-production basis. A nickel-molybdenum steel (carbon 0·13–0·18, nickel 1·65–2·00, molybdenum 0·20–0·30, per cent.) best meets all the requirements, but in some cases a triple-alloy 'economy' steel may be used.

HEAT- AND CORROSION-RESISTING MATERIALS

Creep and Fracture of Metals at High Temperatures: Symposium

E. I. BRIMELOW: 'Creep and Fracture of Metals at High Temperatures.' *Metallurgia*, 1954, vol. 50, Aug., pp. 81–6.

A symposium on the above subject was held at the National Physical Laboratory, Teddington, Middlesex, from May 31 to June 2, 1954. The meeting was attended by engineers, metallurgists and physicists from many countries. The papers presented, together with discussion, will be published in extenso, by H.M. Stationery Office. In the interim, the synopsis referred to above records some points which may be of immediate interest to those concerned with the development and use of high-temperature materials.

The subject matter of the papers (of which a full list is given) is reviewed in sections relating, respectively, to practical implications of creep phenomena, factors involved in the creep process, development of creep-resisting alloys, and the mechanism of creep processes.

Effect of Cyclic Overload on Creep Properties of Inconel

R. H. CAUGHEY and W. B. HOYT: 'Effects of Cyclic Overloads on the Creep Rates and Rupture Life of Inconel at 1700°F. and 1800°F.'
Amer. Soc. Testing Materials, Preprint, June, 1954; 30 pp.

In the latter part of 1947, when the programme covered by this report was initiated, only a relatively limited amount of information was available on the creep and creep-rupture properties of alloys considered suitable for service under pressure at temperatures above 1500°F. (815°C.). The tests reported were made in view of the possibility that Inconel might be a more suitable material than 25-20 chromium-nickel steel for use in re-former furnace tubes, for which more exacting service conditions were then being introduced. The International Nickel Company carried out a series of tests, under constant conditions, at temperatures up to 1800°F. (980°C.), and the M. W. Kellogg Company, with which the present authors are associated, extended the research to determination of the influence of overload, such as might occur in the course of general overheating of the tubes or the development of hot spots, resulting in metal temperatures and thermal stresses in excess of those on which design for such components is based. The two series of tests were integrated; the same heat of Inconel was used in both laboratories, and the values found in the constant-temperature tests formed the basis for assessment of the effects of cycling.

The composition of the material used is given as follows:— nickel 77·73, chromium 14·00, iron 7·29, copper 0·43, carbon 0·05, silicon 0·21, manganese 0·26, sulphur 0·007, per cent. Heat-treatment consisted in holding at 2050°F. (1120°C.) for two hours,

followed by air-cooling. The resulting grain size was A.S.T.M. No. 1 or larger.

Conditions for the individual tests were chosen to determine the effect of thermal and mechanical overloads on 'life', with particular reference to the following aspects:—

(1) *Effect of overstress*, on creep rate and creep-rupture time obtained for steady-state testing conditions. In this section the conditions were adjusted to indicate the effects of (*a*) various levels of overstress; (*b*) various durations of overloading; and (*c*) repeated short applications of overstress, as compared with a single application of the same overstress for the same total period.

(2) *Effect of overtemperature*, on creep rate and creep-rupture time, under steady-state testing conditions: (*a*) influence of time; (*b*) effect of repeated short applications, as compared with a single application of the same temperature for the same total time; (*c*) effect of overtemperature applied with various levels of base load.

Test conditions and individual series of test data are recorded in detail.

Although the number of tests was limited, the results gave consistent indication of the mode of behaviour of coarse-grained Inconel at 1800°F. (980°C.), under conditions of excessive load and/or temperature. The authors draw the following conclusions:—

'Single applications of overloads, for time intervals up to 75 per cent. of the rupture life under the overload condition, have no appreciable damaging effect on the total rupture life of the material, as estimated under the basis conditions. Creep rates after overloading are greater than the initial creep rates, apparently roughly in proportion to the duration of the overload.

'If the overstress is applied in repeated cycles of short duration instead of a continuous period of time, the effects of overstressing become very detrimental. This effect was manifest in every combination of level of load and duration of overstress investigated. Further, creep rates increase after the application of each cycle of overstress; cyclically overstressed specimens failed at much higher creep rates and lower elongations than those which were subjected to a continuous period of overstress for the same total length of time.

'Overtemperatures affect the rupture life of the material in a different manner. Even a single application of overtemperature has a consistently damaging effect on total creep-rupture life. The tests made were too few to indicate whether this damaging effect would tend to increase with the percentage of life spent at the overtemperature.

'If, instead of a continuous period, the overtemperature is applied in short, consecutive intervals, a further decrease in total life appears to take place. This effect is not, however, very significant: the cumulative time spent at overtemperatures has a much more pronounced effect than the number of cycles in which such overtemperature is applied.

'Overtemperature, like overstressing, raises the second-stage creep rate of the material, once the overtemperature is removed and the specimen is permitted to return to the base temperature. For overtemperatures cyclically applied this effect is progressive and cumulative: the basic creep rate will increase after the completion of each style. No clear trend could, however, be discerned with regard to the effect of the number of cycles of overtemperature on total elongation of the material before failure.'

Cyclic Temperature and Load Tests on Austenitic Nickel-Chromium Steel

G. V. SMITH and E. G. HOUSTON: 'Experiments on the Effects of Temperature and Load Changes on Creep-Rupture of Steels.' *Amer. Soc. Testing Materials, Preprint*, June, 1954; 20 pp.

This paper is another of the series on the effect of cyclic high-temperature conditions, presented at the recent meeting of the American Society for Testing Materials.

The steels tested were of the following types:—

Type	C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %	Ti %
Cr-Mo	0·12	0·13	0·35	—	—	—	2·30	1·15	—
18–8 Cr-Ni (Low-C)	0·03	0·54	0·65	0·019	0·010	10·22	19·50	—	—
12% Cr-Mo ..	0·08	0·22	0·37	0·021	0·022	0·19	12·14	0·49	—
18-8 Cr-Ni-Ti ..	0·05	0·010	0·53	0·010	0·017	10·81	17·61	—	0·42

Particular attention was given to the properties of the extra-low-carbon steel.

In the initial tests (at 1100° and 1500°F. (593° and 815°C.) the final creep-test load was attained by ten equal increments or decrements, at 48-hour intervals, over a period of 480 hours. Comparison was made with behaviour under constant stress. All tests were run for 2500–3000 hours total time.

Tests were also made in which the temperature was continuously cycled to 50°F.±5°F. (28°C.±3°C.) above and below the mean test temperature, at a frequency of 0·5 cycle per hour: rate of temperature change was constant except immediately at the extremes of temperature. The periodic strain measurements were taken only as the cycling temperature passed through the mean: thermal length changes were therefore not involved. Comparison was made with results of constant-temperature tests at the mean temperature of the cycle.

On the 18-8 low-carbon steel a series of creep-rupture tests was made in which either the specimen

was cooled to room temperature once a week while the specimen remained under load, or the load was removed from the specimen once a week for 24 hours, while the temperature remained constant. The results are compared with those of uninterrupted tests.

The observations made led to the following conclusions:—(1) Either upward or downward step loading during the first 480 hours of 3000-hour creep tests of 2¼ per cent. chromium-molybdenum steel at 1000°F. (538°C.) resulted in approximately twice the creep rate found in the constant-load test. Similar tests on the low-carbon 18-8 chromium-nickel steel showed that creep rate was slightly lowered by either upward or downward step loading at 1100°F. (593°C.), but that at 1500°F. (815°C.) either type of loading raised the creep rate somewhat above that observed in constant-load tests. The effects were, however, of relatively small magnitude when considered in terms of creep strength, i.e., stress required to cause a specific rate of creep. In no case did the difference exceed 15 per cent.

(2) Cycling the temperature by 50°F. (28°C.) above and below the nominal test temperature appears to cause an abnormal or increased rate of creep at relatively high temperature, but not at relatively low temperature. Thus, a significant effect of cycling was observed in 18-8 chromium-nickel at 1500°F. (815°C.), but not at 1100°F. (593°C.); in the high-chromium steel at 1300°F. (704°C.), but not at 1000°F. (538°C.), and in 18-8-Ti at 1300°F. (704°C.) but not at 1100°F. (593°C.).

(3) In the low-carbon chromium-nickel steel lowering the temperature once a week to room temperature had little, if any, effect on minimum creep rate or rupture time at 1100°F. (593°C.), but resulted in significant decrease in creep rate (increase in creep strength) at 1500°F. (815°C.), and a slight increase in time for rupture (increase in rupture strength) at the same temperature.

(4) With the low-carbon 18-8 chromium-nickel steel removal of the load for 24 hours once a week had little effect on creep rate at 1110°F. (593°C.), but resulted in a pronounced increase in time to rupture (which corresponds, however, to only slight increase in rupture strength). In a similar test at 1500°F. (815°C.) there was no significant effect on time to rupture, but a pronounced decrease in creep rate (increased creep strength).

Notch-Sensitivity in Fatigue of Nickel-Cobalt-Chromium-Iron-base Alloy

P. R. TOOLIN: 'Influence of Test Temperature and Grain Size on the Fatigue Notch-Sensitivity of Refractaloy 26.' *Westinghouse Research Laboratories, Scientific Paper* 1834, May 14, 1954; 22 pp.

The material studied was of the following type: nickel 35·4, cobalt 21·4, chromium 18·5, molybdenum 2·99, titanium, 2·63, aluminium 0·14, silicon 1·13, manganese 0·86, per cent., balance iron. The work described is an extension of an earlier investigation dealing with factors which influence the fatigue strengths of un-notched specimens of the same material.

Heat-treatments, with details of grain-size and hardness produced by them, are given below:—

Heat-Treatment					Grain-Size, A.S.T.M. No.	Diameter, mm.	Average Hardness, D.P.N.
Solution Temperature		Time,	Ageing. Hours at				
°F.	°C.	min.	1500°F. (815°C.)	1350°F. (735°C.)			
1800	980	20	—	44	7·5	0·027	345
1800	980	20	20	20	7·5	0·027	310
2100	1150	60	20	20	3·5	0·107	300

All three treatments resulted in materials the hardness of which increased during the tests at 1050°F. (565°C.) and 1200°F. (648°C.), and decreased during tests at 1500°F. (815°C.) and 1600°F. (870°C.).

The fatigue tests were of the repeated-bend type, made with completely reversed loading at 7200 c.p.m.

The conclusions drawn from the tests are summarized below:—

(1) Over the temperature range 80°–1500°F. (26·8°–815°C.) the notch-sensitivity in fatigue of Refractaloy 26 increases as the mean grain diameter falls from 0·11 to 0·027 mm. (the full range investigated).

(2) The fatigue strengths, of both notched and un-notched specimens of materials (*a*) solution-treated at 1800°F. (980°C.) and aged at 1350°F. (735°C.), (*b*) solution-treated at 1500°F. (815°C.) and aged at 1350°F. (735°C.), and (*c*) solution-treated at 2100°F. (1150°C.) and aged at 1500° and 1350°F. (815° and 735°C.) reach maxima between 1100°F. (593°C.) and 1200°F. (648°C.). For material of both grain-sizes studied maximum notch-sensitivity occurs at the temperature of maximum fatigue strength.

(3) The presence of a stress concentration lowers the temperature at which the fine- and coarse-grained materials have equal fatigue strengths.

(4) For the alloy having a mean grain-size of A.S.T.M. 7·5 (obtained by solution treatment at 1800°F. : 980°C.), variation in ageing treatment has an appreciable effect on fatigue strength at 1200°F. (648°C.), but a negligible influence on notch-sensitivity.

Scaling and Cracking-Corrosion in High-Temperature Alloys

P. GALMICHE: 'The Problem of Sealing and Cracking Corrosion in High-Temperature Gas-Turbine Alloys.' *Recherche Aéronautique*, 1954, May-June, pp. 45–53.

The author reports research carried out in the laboratories of the Office National d'Etudes et de Recherches Aéronautiques (O.N.E.R.A.).

The object of the tests made was two-fold: (1) to determine the scaling-resistance of a series of alloys of iron-, nickel-, or cobalt-base types, in gaseous atmospheres simulating those normally encountered in gas-turbine operation; (2) to study the mechanism of cracking-corrosion, with particular reference to chromium-enrichment or chromium-impoverishment of the surface of the alloys.

The materials tested included nickel-chromium-base alloys of the Nimonic series, A.T.G.S., and Per 2 types; 18-8 chromium-nickel steel; Hastelloy B (nickel-molybdenum-base alloy), and Oneral (cobalt-chromium-base alloy). For investigation of the influence of chromium content of the surface layers, some of the alloys were chromium-enriched, others chromium depleted, by special treatments, details of which are given.

In the scaling tests most of the exposures were made in the range 700°–900°C. and the normal duration was 100 hours; some supplementary tests were made at 1000°C. and for longer periods. The fuel used was in most cases kerosene having a sulphur content limited to the 0·5 per cent. maximum required by aeronautical specifications, but some additional tests were made in combustion gases formed from a fuel of higher sulphur content. Scaling-resistance was assessed by the weight of metal lost, and by micrographic examination of the specimens.

The observations made indicate that, provided that a high-grade fuel is used, scaling presents no problem in the case of high-temperature materials currently used in gas turbines.

The second section of the investigation was concerned with the penetration of corrosion and corrosion-cracking arising from exposure of alloys to contaminated atmospheres of both oxidizing and reducing types; particular attention was given to the difference in severity of attack in oxidizing and reducing atmospheres. Numerous typical examples of penetration and associated corrosion-cracking are illustrated. The rôle of chromium as a protective element is demonstrated by reference to the superior resistance to attack of the alloys which had been treated to increase the chromium content of the surface layers.

Machining Tests on Nimonic 90 (Nickel-Chromium-Cobalt-base Alloy)

B. BUTTERFIELD: 'Machinability of Nimonic 90, with Special Reference to Hi-Jet Lubrication and CO_2 Coolant and their Effects on Tool Wear.' *Aircraft Engineering*, 1954, vol. 16, Sept., pp. 341–2.

Abstract of thesis presented for diploma of the College of Aeronautics, Cranfield.

'The purpose of the investigation was to examine fundamental problems concerned in machining this nickel-base heat-resisting alloy. Among the tests is one designed to investigate the possibility of increasing the life of a tungsten-carbide-tipped tool, by using the Hi-Jet high-pressure lubrication system, and carbon-dioxide cooling of the tool tip.

'A method of obtaining a comparative tool-life test without complete failure of the tool is described, based on a flank 'wearland' of 0·030 in. as being the end of tool-life for all practical purposes. The optimum tool angles were used throughout and a constant depth of cut of 0·10 in.

'It was found that speeds of 100–125 ft./min. gave a maximum tool-life at all feeds up to 0·020 in./rev. (Higher feed-rates are beyond the capacity of the machine.) An optimum feed-rate prevailed at 0·015 in./rev., above which value tool-forces are considerably increased and tool-life is shortened. (The special lathe and CO_2 cooling apparatus installed for this work are illustrated).

'Use of the Hi-Jet type of apparatus resulted in increases of up to 200 per cent., but using the normal overhead application of oil or cutting fluid no increase in tool-life could be obtained. Results when using carbon dioxide as a coolant were noteworthy; a 400 per cent. increase in tool-life was obtained.

'Although a more comprehensive test is required before the evidence is conclusive, it would appear that CO_2 cooling may provide the answer to machining operations where high temperatures are generated.'

Comparison of machining in various conditions is made by means of a graph showing results of (*a*) cutting dry, and (*b*) cutting with high-pressure lubrication and CO_2 coolant: additional data are provided in tables from a diploma thesis by J. T. D. HOLT (*ibid.*, p. 342), which also refers to tests on Nimonic alloys machined under the two conditions.

Nimonic 90 Spring Alloy

HENRY WIGGIN AND CO., LTD.: 'Nimonic 90 for Springs.' *Publn.* 764*, 1954; 4 pp.

Nimonic 90, a nickel-chromium-cobalt-base alloy, meets the need for a spring material which will operate satisfactorily, at reasonable stresses, at temperatures higher than those for which the nickel-chromium-iron alloy Inconel can be recommended. Nimonic 90 has the further advantage that it is highly resistant to attack by a number of corroding media which could accelerate failure by fatigue.

This publication gives information on physical and electrical properties: tensile and torsion properties, at temperatures up to 800°C. of the alloy in bar form; mechanical properties, at room temperature, of 14 s.w.g. wire. Recommendations are made with regard to condition of wire, coiling and ageing procedure, and maximum design stresses for round wire compression springs of Nimonic 90 operating at various temperatures.

Properties of Nickel-containing Borides and Aluminides

J. L. EVERHART: 'New Refractory Hard Metals.' *Materials and Methods*, 1954, vol. 40, Aug., pp. 90–2.

This article describes four materials of the cermet

* *We shall be pleased to supply a free copy of this publication.*

type: zirconium boride, chromium boride, molybdenum-nickel boride, and nickel aluminide. Properties are tabulated, and potential uses of the respective materials, in chemical and/or high-temperature engineering and other fields, are reviewed.

With regard to the nickel-containing materials mentioned, the following comments are made:—

Molybdenum-Nickel Boride. This material has been found to show excellent resistance to dilute sulphuric acid. As a cutting-tool material it also shows considerable promise: for cutting aluminium and brass it is claimed to compare favourably with high-speed steels and cemented carbides. Further applications for this type of boride are likely to develop from its corrosion- and abrasion-resisting characteristics, e.g., use in spray-nozzle inserts, die or mould liners, and plungers for automatic presses for production of small parts from highly abrasive materials. It also shows promise as a brazing material, for use on electronic components in vacuum tubes and magnetrons.

Nickel Aluminide. Commercial grades of this type of material show good elevated-temperature stress-rupture properties, combined with better ductility than is usually found in cermets. Impact-resistance of nickel aluminide is also an interesting feature which, together with ability to withstand thermal-shock and to retain a good degree of mechanical strength at temperatures up to about 2100°F. (1150°C.) will ensure consideration of this material for several applications in high-temperature service. Tests to assess its suitability for stator blades or guide vanes in jet engines are currently under way.

Nickel Additions to Molybdenum Disilicide

H. A. DE VINCENTIS and W. E. RUSSELL: 'Additives and Molybdenum Disilicide.' *Nat. Advisory Committee for Aeronautics Report R.M. E54B15*, May, 1954; 22 pp.

This report covers investigations of the effects of adding approximately 6 per cent. of nickel, cobalt, or platinum, on some properties of molybdenum disilicide. Such additions were found to lower the modulus of rupture and lessen resistance to oxidation at high temperatures. They did not affect the resistance of the molybdenum disilicide to thermal shock.

Extrusion of Nickel-containing Steels and Alloys

'The Ugine-Sejournet Process for the Hot Extrusion of Steel.' *Machinery* (*Lond.*), 1954, vol. 85, Sept. 3, pp. 471-80.

Considerable interest has been aroused during recent years, in the Ugine-Sejournet extrusion process, principally on account of the novelty of using glass as the lubricating medium. By this technique it has been found possible not only to extrude lower-alloy steels in greatly increased lengths, but also to process higher-alloy materials, such as corrosion- and heat-resisting steels and alloys, for which this method of working was previously impracticable. (Apart from the fundamental change in introduction of glass as the lubricant, the Ugine-Sejournet process is broadly similar to conventional extrusion procedure.) Various methods are adopted for applying the glass to the billet, the die, and the mandrel, and the types of glass used are suited to the requirements of the individual materials and the extrusion operations involved.

This article, which is based on three papers recently published by engineers associated with the development, gives a detailed review of the history of the process, factors affecting the actual extrusion operation, and the advantages of the Ugine-Sejournet technique, from the technical and economic points of view. Among the advances which have resulted from its introduction is the possibility of extruding tubes and other forms in materials which could not previously be hot-pierced. Foremost among these are the high-nickel alloys of many types which have been developed for gas-turbine components and other applications in high-temperature engineering.

Production of Stainless-Steel Compressor Blades for Gas Turbines

'Precision Forging Stainless-Steel Compressor Blades for Gas Turbines.' *Machinery* (*Lond.*), 1954, vol. 85, Aug. 27, pp. 419-29.

One of the major problems encountered in the construction of gas turbines is the production, to very close tolerances, of the large numbers of blades required. A considerable amount of research has been carried out by industry in order to establish the most efficient techniques: methods in current use include machining from solid billets, machining from forgings produced to commercial standards of accuracy, precision casting by the lost-wax process, and precision forging. In the works of the Bristol Aeroplane Company each of these methods is being used as occasion requires, but particular attention has been given to precision forging, after which operation only a small amount of machining and finishing is necessary. This process has the further advantage that there is relatively small wastage of expensive material, and that greater latitude can be offered to the designer in specifying blade forms which will ensure the most efficient working in the completed gas turbine. The technique of precision forging is described in the present article, which is amply illustrated in the manner characteristic of production-practice articles prepared for *Machinery*.

Stator and rotor blades ranging in overall length from 2 to 12½ in. are being made by this method, mainly from two types of material: (1) stainless steel to D.T.D. 282 (nickel 6–10, chromium 17 per cent.), and (2) a steel containing nickel 3–4, chromium 12·5–14·5, per cent.

The final section of the article contains particulars of methods used in production and servicing of the rough-forging, pre-forming, and finishing dies used in the various operations previously described. The dies are made from a nickel-chromium-molybdenum steel to B.S. specification, hardened to 500 Brinell. *Inter alia*, spark-machining equipment used for reconditioning forging dies is described.

Effects of Nuclear Radiation on Constructional Materials

See abstract on p. 188.

Specification for Metallic Electrical-Resistance Materials

See abstract on p. 188.

Nickel Alloys for Brazing of Sintered Molybdenum

K. C. DIKE: 'Evaluation of Alloys for Vacuum Brazing of Sintered Wrought Molybdenum for Elevated-Temperature Applications.'
Nat. Advisory Committee for Aeronautics, Tech. Note 3148, May, 1954; 13 pp.

Although considerable data exist in the literature on the joining of molybdenum, by several welding methods, most of the welds made have proved to be unsatisfactory, due to porosity, cracks, or impurities in the weld. Recrystallization in the heat-affected zone has been a major problem, in view of its effect in promoting embrittlement. Little work has yet been done on brazing of molybdenum with high-temperature alloys, but results of recent research on the use of high-melting-point alloys for this purpose appeared encouraging, and have been extended by the work described in the present paper.

The programme covered (*a*) fabrication of binary and ternary alloys having liquidus temperatures in the range 2000°–2500°F. (1095°–1370°C.) approx.; (*b*) evaluation of the brazing characteristics of these alloys, with molybdenum, in vacuum; (*c*) determination of the tensile strengths, at room temperature and 1800°F. (980°C.), of butt-brazed molybdenum joints. Twenty-five binary and ternary alloys were prepared, and the brazing characteristics of these materials, together with those of three commercial alloys, were evaluated. Nominal compositions used are shown below:—

Nominal Composition of Brazing Alloys, per cent.

*Fe 50/Pd 50	Au 40/Ag 40/Pd 20
*Pd 60/Cu 40	Pt 60/Cu 40
*Nb 52/Ni 48	*Fe 87/Ti 13
*Ni 80/Mn20	*Fe 84/Zr 16
*Ni 84/Ti 16	Pd 50/Ag 50
	Ni 50/Sn 50
	Fe 58/Al 42
*Pd 70/Cu 30	Fe 50/Si 50
*Pd 80/Co 20	Mn 60/Fe 40
*Pd 80/Ni 20	Cu 65/Ni 35
*Pd 60/Ni 40	Cr 52/Ni 48
*Pd 70/Fe 30	Al 45/Ni 35/Cu 20
*Ni 76/Nb 24	Ni 50/Cu 40/Al 10
	Ni 42/Fe 38/Ti 20

The three commercial alloys were Hastelloy B (nickel-molybdenum-iron), Stoody 6 (cobalt-chromium-base) and Nicrobraz (nickel-chromium-boron).

The room-temperature tensile strengths of butt-brazed joints made with the 13 alloys showing the most promising characteristics (indicated by an asterisk in the table) ranged from 8450 to 49,400 p.s.i. (3·5 to 22 tons per sq. in.). The ten alloys which gave joints having room-temperature strengths above 21,000 p.s.i. (9·5 tons per sq. in.) were tested at 1800°F. (980°C.), at which temperature strengths of 0 to 19,000 p.s.i. (8·5 tons per sq. in.) were obtained.

The three alloys which, in the high-temperature tests, showed bonding strengths of 17,000–19,000 p.s.i. (7·5–8·5 tons per sq. in.) were considered satisfactory, and were heated for 24 hours at 1800°F. (980°C.) in vacuum to determine the extent of diffusion and the effect on tensile strength. Two of these alloys, containing respectively nickel 84, titanium 16, per cent., and niobium 53, nickel 48, per cent., appeared to be unaffected by such exposure, and it is considered that these types may be potentially useful for brazing molybdenum, for high-temperature applications. The alloy containing palladium 50, iron 50, per cent. deteriorated under high-temperature exposure: strength at 1800°F. (980°C.) which was originally 17,000 p.s.i. (7·5 tons per sq. in.) dropped to 7,000 p.s.i. (3 tons per sq. in.).

Resistance Welding of High-Alloy Materials used in Jet Engines

F. J. WALLACE: 'Resistance Welding in Jet Engine Manufacturing.' *Welding Jnl.*, 1954, vol. 33, Aug., pp. 762–7.

The article describes, with many detailed illustrations, equipment and methods employed in the plant of Pratt and Whitney Aircraft. Materials welded by such techniques include austenitic and martensitic stainless steels, low-alloy steels, and nickel- and cobalt-base alloys.

Directory of British Hardfacing Electrodes

See abstract on p. 189.

Internal Nitriding of Austenitic Nickel-Chromium Steel Tubes

F. BRAUMANN and H. KRÄCHTER: 'Nitriding of Austenitic Chromium-Nickel-Molybdenum Steel Tubes by Application of Nitrogen under Pressure, at 700°C.' *Archiv f.d. Eisenhüttenwesen*, 1954, vol. 25, July-Aug., pp. 373–5.

The authors report examination of titanium-stabilized 18-10-2 chromium-nickel-molybdenum steel tubing, the internal surface of which had been exposed to high-pressure nitrogen for 10,000 hours at 700°C. This treatment had increased the nitrogen content of the steel from 0·017 per cent. (as shown by analysis of the steel before nitriding) to 2·2 per cent. The nitrided layer was brittle and showed some hair cracks. In it there occurred, in association with the austenitic matrix, two nitrides, one a mixed iron-chromium type (Fe, Cr)N, and the other the almost pure ε-phase iron nitride Fe_2N.

Wear-Corrosion Tests in High-Temperature Water

R. C. WESTPHAL and J. GLATTER: 'Wear and Corrosion of Materials in High-Temperature Water.' *Materials and Methods*, 1954, vol. 40, Aug., pp. 100–1.

This note, condensed from a paper presented before the American Society of Mechanical Engineers, records data obtained (in the laboratories of Westinghouse Electric Corporation) by a wear test simulating service involved in nuclear power plant designed for the propulsion of a submarine, in which many mechanical parts operate under unusual conditions. Antifriction bearings, linear sliding guide bearings, nuts and bolts are required to function in high-purity high-temperature water and, since there is no lubricant present except the water, choice of suitable materials is governed by consideration of resistance to corrosion as well as to wear.

In the experiments reported in this paper, various material combinations were tested, in oxygenated and (in some cases only) hydrogenated water. Results are tabulated for each combination of materials studied. Among the general conclusions drawn are the following:—

'Nitrided surfaces had the greatest wear-resistance and antigalling characteristics, but nitrided chromium plating and nitrided titanium were the only materials which had sufficient corrosion-resistance in water to be usable.

'The next best material in wear-resistance was hard industrial chromium plate, which could be used with a wide variety of mating materials. Corrosion-resistance was assured by plating on a base of stainless steel. No other corrosion-resisting material was found which approached the wear-resistance of this plate.

'Excellent wear-resistance was found with straight high-chromium steels, as compared with the austenitic nickel-chromium grades, but the former type was unsuitable for use in oxygenated water, due to pitting corrosion. Of the austenitic steels, only the age-hardenable grades were usable, even to a limited extent.

'Surface finish can affect wear-resistance and tendency to galling. When the mating materials differ greatly in hardness it is important that the harder of the two shall have a good finish, otherwise wear is accelerated by continuous cutting action on the softer material or by loading of the rough surface followed by abrasive wear. Under certain circumstances wear can be reduced by having a controlled degree of roughness on one of the mating surfaces.

'Comparison of the wear factors in (*a*) oxygenated and (*b*) hydrogenated water indicates the important influence of corrosion on wear-resistance: the greatest difference is observed with the materials most susceptible to oxidation. Silicon bronze *vs.* chromium plate showed a very low wear factor in hydrogenated water and a very high one in oxygenated water, and there was an 88 per cent. reduction in wear, in the hydrogenated water, for a KR Monel *vs.* chromium plate combination. Stellite combinations, however, which were inherently more resistant to oxidation, did not show so great a difference.'

Materials used in Equipment for Storage and Transport of Milk and Beer

J. D. USHER: 'Chemical Engineering Methods in the Food Industry.' *Chemistry and Industry*, 1954, Aug.21, pp. 1031–9.

The information given in this paper, although broadly applicable to many branches of the food industry, relates specifically to practice in handling milk and beer. It is pointed out that the problems involved in equipment for such applications are two-fold: there is (1) the need for operating conditions satisfying the requirements of the processes concerned, and (2) the necessity of ensuring that all equipment complies with generally accepted standards of sanitary design, which are drawn up to facilitate maintenance of plant in a clean and sterile condition. Design and materials employed to satisfy these requirements are discussed, with typical examples of plant, and of processing and storage equipment.

Transport of milk is dealt with at some length, being typical of methods adopted to prevent spoilage of a raw perishable liquid. Practice is described as affecting three stages of movement: from farm to collecting centre, from collecting centre to processing depot, and internal transport within the factory.

In dealing with brewing industry, the varying conditions involved in the fermenting, conditioning and storage are described.

Rail and road transport of both milk and beer is separately considered, with emphasis on the additional problems of weight reduction, strength, and rigidity involved.

Attention is directed to the many uses of stainless steel, of the nickel-chromium and nickel-chromium-molybdenum types, in both the dairy and the brewing industry, and to the importance of these materials and of aluminium alloys and glass, in ensuring the clean and sterile conditions essential for maintenance of quality in the substances handled in all branches of the food industries.

Monel Filters in Paper Mills

See abstract on p. 192.

Determination of Niobium in Stainless Nickel-Chromium Steels

G. W. C. MILNER and A. A. SMALES: 'The Determination of Niobium in Stainless Steel.' *Analyst*, 1954, vol. 79, July, pp. 425–30.

The authors describe a procedure in which niobium is first separated from many of the other constituents of the steel, by precipitation with tannic acid and cinchonine, from a slightly acid solution. Silica is used as a carrier for the niobium precipitate, and after filtration and ignition the silica is removed as its volatile fluoride. The oxide residue is next fused with potassium bisulphate, and the melt is dissolved by extraction with an aqueous solution of tartaric acid. The concentration of niobium is finally determined absorptiometrically, use being made of the yellow colouration produced with potassium thiocyanate. For accurate work it is necessary to incorporate niobium-95 tracer, to correct for small losses

of niobium occurring in the chemical separation, but the results for niobium in the absence of the radioactive tracer are usually sufficiently accurate for satisfactory quality control. Determination of tantalum, in the separated earth-acid oxides, by radioactivation, is also briefly discussed.

Determination of Chromium in Alloy Steels and Copper-base Alloys

See abstract on p. 196.

Electrographic Identification of Steels and Alloys

See abstract on p. 189.

PATENTS

Extraction of Nickel

Nickel is extracted (as an iron-nickel alloy) from low-grade oxide or silicate ore by mixing successive charges of molten ore with molten iron and periodically withdrawing the iron-nickel alloy produced. Further iron is added as required, and iron oxide formed may be reduced by adding a suitable reducing agent, preferably one resulting in an exothermic reaction. The molten ore may be superheated before addition. Mixing of metal and ore may be effected by air-blowing, preferably in a converter.

SOCIÉTÉ D'ELECTRO-CHIMIE, D'ELECTRO-MÉTALLURGIE ET DES ACIÉRIES ELECTRIQUES D'UGINE. *Brit. Pat.* 713,713.

One part of material containing nickel is digested with three parts of an ammonia-ammonium carbonate solution, in the presence of an oxidizing gas containing oxygen, until ferrous iron is oxidized to insoluble iron hydroxide. The digested mixture is then leached in a much larger volume of the leach solution, while an oxygen-containing oxidizing gas is fed in. Most or all of the metallic nickel is thus dissolved and is recovered from the leach solution after separation from the residual solids. Nickel may be extracted from oxidic substances in this way if the initial material is first reduced, e.g., in hydrogen at 400°-600°C.

SHERRITT GORDON MINES, LTD. (inventor, F. A. FORWARD). *Brit. Pat.* 715,243.

Production of Clad Metal for Use in Thermionic Valves

Material stated to be suitable for grids and anodes of thermionic valves consists of a nickel base coated first with iron and then with aluminium, the iron and aluminium being in intermetallic association. The iron (10-20μ thick) is preferably applied by warm-rolling, and the aluminium (forming 5-10 per cent. of the total thickness of the sheet) by cold- or hot-rolling. The clad sheet is finally heated at 500°-1000°C. to effect bonding.

TELEFUNKEN GESELLSCHAFT FÜR DRAHTLOSE TELEGRAPHIE, m.b.H. *Brit. Pat.* 710,968.

Nickel Cathodes for Thermionic Valves

Cathodes for thermionic valves consist of a base of high-purity nickel, and an emissive coating containing an agent for reducing the oxide coating. Such an addition replaces the activating constituent usually present as an impurity in the nickel. Preferably about 1 per cent. of zirconium hydride is mixed with the carbonate coating.

STANDARD TELEPHONES AND CABLES, LTD. (inventors, A. H. W. BECK and J. C. JONES). *Brit. Pat.* 710,648.

Rare-Earth Additions to Nickel for Electrodes of Thermionic Valves

High-purity nickel or cobalt for thermionic-valve electrodes is deoxidized by adding to the melt 1-10 lb. of Mischmetall per 1300 lb. of nickel or cobalt, to give a residual content of less than 0·2 per cent. of any one rare-earth metal, in particular less than 0·2 per cent. each of cerium and of lanthanum.

BRITISH DRIVER HARRIS CO., LTD. *Brit. Pat.* 713,063.

Descaling of Nickel and Nickel Alloys

Oxide scale is removed from surfaces which are resistant to the action of alkalis, by immersion in an alkaline fused-salt bath in which sodium fluoride and sodium carbonate form at least 90 per cent. by weight and the ratio $NaF:Na_2Co_3$ is between 4:1 and 1:4. The bath is preferably used at 1500°-2000°F. (815°-1095°C.), and the heating thus involved may be utilized as an annealing treatment, after which the material may be brightened by pickling for a short time in an acid solution. The procedure claimed is suitable for descaling of wrought nickel, high-nickel alloys containing chromium, molybdenum, copper or iron, austenitic nickel-chromium steels, and other steels.

MOND NICKEL CO., LTD. *Brit. Pat.* 713,909.

Purification of Nickel-Plating Baths

Impurities such as arsenic, antimony, bismuth, chromium, iron, lead, copper, silver are removed from nickel-, zinc-, or cadmium-plating baths by absorption on Al_2O_3.

DR. HESSE & CIE. (inventor, A. KUTZELNIGG). *German Pat.* 911,688.

Electrodeposition of Tin-Nickel Alloys

Tin-nickel alloys are electrodeposited from baths in which the only anions are sulphates and fluorides. Example: $NiSO_4.7H_2O$, $SnSO_4$ and NH_4F (or NaF).

J. IRELAND (inventor, A. E. DAVIES). *Brit. Pat.* 714,462 (addition to *Brit. Pat.* 661,154).

Immersion Coating of Metals and Non-Metallic Materials

A coating of ruthenium, rhodium, osmium, iridium, platinum, iron, nickel, cobalt, chromium, aluminium, magnesium, or manganese is formed on metal, or on glass or other non-metallic surfaces, by treating the

basis material with tin chloride solution, subsequently dipping it into a solution of a palladium salt, to form a very thin palladium layer, and finally treating the palladium-coated surface with a solution of a salt of the desired final coating metal. This last solution preferably contains also a reducing agent.

SVENSKA AKTIEBOLAGET GASACCUMULATOR. *Brit. Pat.* 712,687.

Electrode Sealing Plugs of Iron-Nickel-Cobalt Alloy

The electrode of a sparking plug is held in position by a sealing plug made from an iron-nickel-cobalt alloy having a coefficient of thermal expansion equal to that of the insulator body. An example cites an alloy containing iron 54, nickel 29, cobalt 17, per cent., used with a ceramic insulator.

L. J. BROIS. *Brit. Pat.* 715,153.

Nickel in Precious-Metal Contact Alloys

A contact alloy for use in telecommunication apparatus contains 3–10 per cent. of nickel, 0·5–4 per cent. of at least one metal which raises the recrystallization temperature (e.g., copper, cobalt, iron, chromium, manganese), balance gold.

SIEMENS UND HALSKE, A. G. (inventors, E. DÖRING and H. G. MÜLLER). *German Pat.* 910,839.

Heat-Treatment of Complex Nickel-base High-Temperature Alloys

Alloys containing chromium 10–25, titanium 1·8–4·0, aluminium 0·5–4, cobalt 0–25, iron 0–10, per cent., nickel balance, with carbon contents determined by the cobalt content, according to curves given in the specification, are heated at 1150°–1250°C. for periods varying from 2–12 hours at 1150°C. to 0·5–4 hours at 1250°C., cooled to 1100°C., held at 1100°–800°C. for at least 2 hours, and finally held at a lower temperature in the range 650°–850°C. for 1–24 hours. The alloys may also contain silicon up to 1, copper up to 2, molybdenum up to 10, tungsten up to 10, niobum/tantalum/vanadium up to 5, zirconium up to 0·3, boron up to 0·05, per cent. The temperature of the second heat-treatment stage is preferably varied according to the cobalt content.

MOND NICKEL CO., LTD. (inventors, W. BETTERIDGE and A. W. FRANKLIN). *Brit. Pat.* 715,140.

Lubricants for Cold-Forming of Stainless Steel

A mixture of soap, water, and copper powder is used as lubricant for cold-forming alloy steels. A particular application is in the extrusion of 18–8 chromium-nickel steels.

COMPTOIR INDUSTRIEL D'ETIRAGE ET PROFILAGE DE MÉTAUX. *Brit. Pat.* 710,613.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*

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